[Geochemical Analysis & Lab]

EXPERIMENT 8: Determination of Fe with Phenanthroline Method

**Preparation**:

* Sample 50 mL (acidified)
* 1,10-phenanthroline monohydrate(C12H8N2·H2O), ammonium acetate (NH4C2H3O2), *sodium acetate (NaC2H3O·3H2O)\**, acetic acid, *potassium permanganate (KMnO4)*, hydroxylamine hydrochloride (NH2OH·HCl), ferrous ammonium sulfate (Fe(NH4)2(SO4)2·6H2O), HCl, H2SO4
* UV-Visible photometer, sample cells (more than two)
* Chemical balance
* Pipet
* Beaker; 500 mL, 250 mL, 100 mL
* Volumetric flask; 100 mL, 1000mL
* Hot plate
* Glass rod, glass beads

**Procedures:**

1. Reagent Preparation
2. c-HCl, containing less than 0.5 ppm Fe.
3. Hydroxylamine solution: Dissolve 10 g NH2OH.HCl in 100 mL water.
4. Ammonium acetate buffer solution: Dissolve 125 g NH4C2H3O2 in 75 mL water. Add 350 mL conc (glacial) acetic acid.
5. *Sodium acetate solution: Dissolve 200 g NaC2H3O·3H2O in 800 mL water*.
6. Phenanthroline solution: Dissolve 100 mg 1,10-phenanthroline monohydrate, C12H8N2·H2O, in 100 mL water. Add a few drops of c-HCl.
7. Stock iron solution (200mg/L Fe solution): Slowly add 20 mL c-H2SO4 to 50 mL water and dissolve 1.404 g Fe(NH4)2(SO4)2·6H2O. Add 0.1M potassium permanganate (KMnO4) dropwise until a faint pink color persists. Dilute to 1000 mL with water and mix; 1.00 mL = 200 μg Fe.
8. Standard iron solutions: Prepare daily for use. Pipet 5, 25, and 50 mL stock solution into 100-mL volumetric flasks and dilute to mark with water ; 1.00 mL = 10, 50, and 100 μg Fe (1, 5, 10 mg/L Fe).
9. Determination of Fe in sample
10. Mix sample thoroughly and measure 50.0 mL into a 125-mL Erlenmeyer flask. Add 2 mL c-HCl and 1 mL NH2OH·HCl solution. Add a few glass beads and heat to boiling.
11. Continue boiling until volume is reduced to 15 to 20 mL.
12. Cool to room temperature and transfer to a 100-mL volumetric flask.
13. Add 10 mL NH4C2H3O2 buffer solution and 4 mL phenanthroline solution, and dilute to mark with water. Mix thoroughly and allow a minimum of 10 min for maximum color development.
14. Do (step 1) to 4)) exactly the same for the standard solutions in step 1-7) and DIW
15. Set the wavelength of the photometer 510nm.
16. Measure the absorbance of the solutions.
17. Subtract the absorbance value of DIW from those of the sample and standards (This is going to be the corrected absorbance A’)
18. Plot concentrations on the horizontal axis and A’ of the standards on the vertical axis. Do the linear regression and obtain an equation.
19. Put the A’ value of the sample into the equation and obtain the Fe concentration (c) of the sample

**Notes:** \* underlined and italic part may be skipped.